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<p>(21) International Application Number: <b>PCT/US96/13561</b> (22) International Filing Date: <b>22 August 1996 (22.08.96)</b> (30) Priority Data: <b>PN 4998</b>      <b>23 August 1995 (23.08.95)</b>      <b>AU</b> (71) Applicant (for all designated States except US): <del>THE PROCTER &amp; GAMBLE COMPANY</del> [US/US]; <b>One Procter &amp; Gamble Plaza, Cincinnati, OH 45202 (US).</b> (72) Inventor; and (75) Inventor/Applicant (for US only): <b>NAKAMURA, Kiichiro</b> [JP/JP]; <b>1-4-124-609, Koyo-cho Naka, Higashinada-ku, Kobe 658 (JP).</b> (74) Agents: <b>REED, T., David et al.; The Procter &amp; Gamble Company, 5299 Spring Grove Avenue, Cincinnati, OH 45217 (US).</b></p>		<p>(81) Designated States: <b>BR, CN, JP, MX, US, European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).</b>  <b>Published</b> <i>With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i></p>
<p>(54) Title: <b>HAIR TREATMENT COMPOSITION</b> (57) Abstract  A hair treatment composition including: (a) from about 0.6 % to about 10 % by weight, of a fatty alcohol; (b) from about 0.01 % to about 15 % by weight, of a silicone conditioning agent; and (c) from about 0.1 % to about 5.0 % by weight, of a monoalkyl trimethyl ammonium salt.</p>		

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## HAIR TREATMENT COMPOSITION

### FIELD OF THE INVENTION

The present invention relates to a hair treatment composition, and in particular to rinse off hair products having improved performance in wet detangling and dry combing of conditioned hair.

### BACKGROUND OF THE INVENTION

Various hair treatment compositions, such as hair rinse agents and conditioning compositions have been used to provide gloss and condition to hair. Such compositions may be used either in conjunction with or following shampooing of the hair. Shampooing the hair cleans the hair by removing excess soil that has built up on the hair due to contact with the surrounding atmosphere. Sebum, which is secreted by the head, is also removed by shampooing. However, the shampooing process generally leaves the hair in a wet, tangled and generally unmanageable state.

In order to alleviate the problem associated with shampooing, a variety of approaches have been developed including the application of hair conditioners post shampooing, or combining a conditioning composition with the shampoo. Such conditioners and hair rinses typically work by depositing a polymeric film, or other material onto the hair. These products have been successful to varying degrees, in leaving the hair manageable, however they have not been completely successful in overcoming the difficulties relating to detangling wet hair and combing or brushing dry hair.

Typically, hair conditioning compositions have a thick creamy rheology making them suitable and desirable for use. Such products are generally based upon surfactants, including a combination of fatty alcohols and quaternary ammonium salts. Both the fatty alcohols and quaternary ammonium salts provide hair conditioning benefits to the hair. However, such materials have not fully met consumer's needs, especially for dry combing performance.

Silicones have also been incorporated into hair treatment compositions such as shampoos and conditioning shampoos in order to deliver a desired level of smoothness to the hair. A number of prior publications such as US Pat. No.

2,826,551, US Pat. No. 3,964,500, US Pat. No. 4,364,837 and US Pat. No. 4,4788,006 disclose the use of silicones in shampoo compositions.

A hair treating composition including a dimethyl silicone rubber and a quaternary ammonium salt is disclosed in US Patent 4,950,468. The quaternary ammonium salt consists of a combination of stearyl- and behenyl trimethyl ammonium chloride. The composition in this disclosure is said to have excellent resistance to washing. The effect of washing however will often leave wet hair tangled and unmanageable despite the application of such hair treatment compositions.

A hair rinse conditioner including a dodecyl triethyl quaternary ammonium compound, fatty alcohol and a cyclic or linear silicone is disclosed in USP 4,818,523. The composition in this disclosure is said to provide conditioning styling ease and manageability of hair, but it does not disclose the performance in wet detangling and dry combing of hair.

It is an object of the present invention to overcome or at least alleviate one or more of the difficulties associated with the prior art.

It is a further object of the present invention to provide a hair treatment composition having improved wet detangling and dry combing capabilities.

#### SUMMARY OF THE INVENTION

The present invention relates to a hair treatment composition including the combination of:

- 0.01 to 15% of a silicone conditioning agent;
- 0.6 to 10% of a fatty alcohol, and
- 0.1 to 5.0% of a monoalkyl trimethyl ammonium salt.

Most preferably the silicone conditioning agent is a polydimethyl siloxane gum having a mass molecular weight of from 200,000 to one million; the fatty alcohol is stearyl alcohol or cetyl alcohol or a mixture thereof; and the monoalkyl trimethyl ammonium salt is behenyl trimethyl ammonium chloride.

The composition provides excellent wet detangling and dry hair combing capabilities following application to the hair.

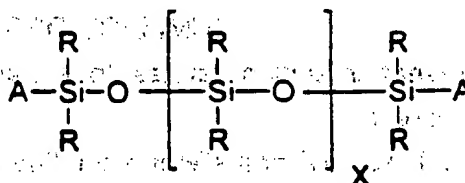
### DETAILED DESCRIPTION OF THE INVENTION

Silicone conditioning agents are known for providing hair conditioning when applied in treatment compositions. Such materials tend not to leave the hair looking "dirty" as typical hair conditioners may. The present invention includes from about 0.01 to 15% of a silicone conditioning agent, preferably from 0.1% to 10%, most preferably from 0.5% to 5%.

Preferably the silicone conditioning agent in the present invention has a molecular weight of from 200,000 to 1,000,000. The silicone conditioning agent useful in the invention most preferably includes a non-volatile silicone however a volatile silicone may also be incorporated as part of the silicone mixture. The term "volatile", as used herein, refers to a silicone conditioning agent that has a measurable vapour pressure.

The non-volatile silicone may be either a polyalkyl siloxane, a polyaryl siloxane, a polyalkylaryl siloxane, an amino siloxane or a polyether siloxane copolymer. Mixtures of these silicones may also be used. The dispersed silicone is preferably insoluble in the hair treatment matrix.

More specifically, materials such as polyalkyl or polyaryl siloxanes may have the following structure:



wherein R is alkyl or aryl, and X is an integer from about 7 to about 8,000. A represents groups which block the end of the silicone chains.

The alkyl or aryl groups substituted on the siloxane chain (R) or the ends of the siloxane chain (A) may have any structure, but preferably result in silicones which remain fluid at room temperature, are hydrophobic, are neither irritating, toxic or otherwise harmful when applied to the hair, are compatible with the other components of the composition, are chemically and storage stable, and are capable of being deposited on, and condition the hair.

Suitable A groups include methyl, methoxy, ethoxy, propoxy, and aryloxy. The two R groups on the silicone atom may represent the same or different groups. Preferably R represents the same group. Suitable groups include methyl, ethyl, propyl, phenyl, methylphenyl and phenylmethyl.

Preferred polyalkyl siloxanes are polydimethyl siloxane and polydiethyl siloxane. Most preferred are polydimethyl siloxanes with viscosities ranging from about 5 to 500,000 centistokes at 25\_C. These siloxanes are available, for example, from the General Electric Company as the "Viscasil" series and from Dow Corning as the "Dow Corning 200" series. The viscosity can be measured by means of a glass capillary viscometer as set forth in Dow Corning Corporate Test Method CTM0004. Preferably, the viscosity ranges from about 350 centistokes to about 100,000 centistokes.

The preferred polyalkylaryl siloxanes that may be used include for example polymethylphenylsiloxanes having viscosities of about 15 to 65 centistokes at 25\_C. These siloxanes are available from the General Electric Company as SF 1075 methyl phenyl fluid or from Dow Corning as 556 Cosmetic Grade Fluid. Additionally poly(dimethyl siloxane) (diphenyl siloxane) copolymers having a viscosity in the range of from about 10 to about 100,000 centistokes at 25\_C are useful.

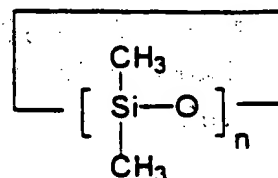
The preferred polyether siloxane copolymer that may be used includes a polypropylene oxide modified dimethylpolysiloxane (eg Dow Corning DC 1748), although ethylene oxide or mixtures of ethylene oxide and propylene oxide may also be used.

References disclosing suitable silicones include US Pat. No. 2,826,551, US Pat. No. 3,964,500, US Pat. No. 4,364,837 and British Pat. No. 849,433. All of these patents are incorporated herein by reference. Also incorporated herein by reference is "Silicon Compounds" distributed by Petrarch Systems Inc. 1984. This reference provides a very good listing of suitable silicone materials.

A silicone conditioning agent found especially useful in the present composition to provide good dry combing is silicone gum. "Silicone gum" materials denote high molecular weight polydiorganosiloxanes generally having a mass

molecular weight of from about 200,000 to about 1,000,000. Suitable silicone gums are described in "Silicon Compounds" by Petrarch (referred to above) and others including US Pat. No. 4,152,416 and Noll, Walter, "Chemistry and Technology of Silicones", New York, Academic Press 1968. Also describing silicone gums are General Electric Silicone Rubber Products Data Sheets SE30, SE33, SE54 and SE76. All of these described references are incorporated herein by reference. Specific examples include polydimethylsiloxane, (polydimethylsiloxane)(methylvinylsiloxane) copolymer, poly(dimethylsiloxane) (diphenyl siloxane) (methylvinylsiloxane) copolymer and mixtures thereof. The gums may contain some minor (eg 6% to 14% of total gum weight) of a cyclic or linear volatile silicone.

The volatile silicone may be incorporated to act a solvent for the silicone gum. Examples of volatile silicone hair conditioning material that may be incorporated have a boiling point in the range of about 99°C to about 260°C and have a solubility in water of less than 0.1%. Preferred volatile silicones may be either a cyclic or a linear polydimethylsiloxane. The number of silicone atoms in the cyclic silicones is preferably from about 3 to about 7, more preferably 4 or 5. The general formula of such silicones is



wherein n is 3 to 7.

The linear polydimethyl siloxanes have from about 3 to 9 silicone atoms and have the general formula:



where n is 1 to 7.

Silicones of the above type, both cyclic and linear, are available from Dow Corning Corporation, Dow Corning 344, 345 and 200 fluids, Union Carbide, Silicone 7202 and Silicone 7158, and Stauffer Chemical SWS 03314.

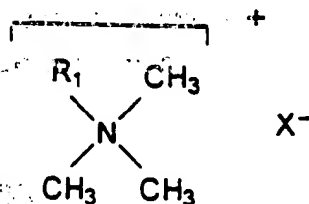
Fatty alcohol materials are desirably included into hair treatment compositions to provide hair manageability benefits, and ease of styling for the user. Suitable

fatty alcohols that are useful as conditioning agents herein are described in "Baileys Industries Oil and Fat Products" (Third Edition D Swen, Ed. 1979), incorporated herein by reference. Further examples of suitable fatty alcohols are disclosed in the following documents, each of which are incorporated herein by reference; US Pat. No. 3,155,591, US Pat. No. 4,165,369, US Pat. No. 4,269,324 and British Specification 1,532,535.

Preferably, the fatty alcohol in the present invention has a melting point higher than 30°C.

Specific examples of fatty alcohol materials include stearyl-, cetyl-, myristyl-, behenyl-, lauryl-, and oleyl alcohols and mixtures thereof. Most preferred of the fatty alcohols are cetyl and stearyl alcohol or mixtures thereof. The composition should include from 0.5 to 10% by weight of the total composition of a fatty alcohol, preferably 2 to 9% and most preferably 3% to 8%. In combination with for example a silicone conditioning agent, and a monoalkyl trimethyl ammonium chloride, it has been found that the hair treatment composition that includes a fatty alcohol provides improved wet detangling and dry combing capabilities.

Monoalkyl trimethyl ammonium salts which may be useful in the present invention have the formula:



where R<sub>1</sub> may be selected from an aliphatic group of at least 14 carbon atoms, and X is an anion selected from halogen, acetate, phosphate and alkyl sulfate radicals. The aliphatic groups may contain, in addition to carbon and hydrogen atoms, ether linkages as well as amido groups among other groups.

The preferred monoalkyl trimethyl ammonium salt ingredient is behenyl trimethyl ammonium chloride. Preferably, it is present in an amount of from 0.1 to 5% by weight based on the total weight of the composition.



The majority of the remainder of the composition is made up of water. It is generally present at a level of from about 20% to 98%, preferably from about 60% to 95%, more preferably from 80% to 90% content of the total composition.

The hair treatment composition may also include a variety of other components suitable for rendering such compositions acceptable for use. Such components are generally well known to those skilled in the art and may include for examples preservatives such as benzyl alcohol, trimethyl parabin, propyl parabin and imidazolidinyl urea, thickeners and viscosity modifiers such as a hydroxy ethyl cellulose and xantham gum, pH adjusting agents such as citric acid, sodium citrate, succinic acid, phosphoric acid, sodium hydroxide, sodium carbonate etc; perfume, dyes and sequestering agents such as disodium ethylene diamine tetraacetate. Such agents generally are used individually at a level of from about 0.01% to about 10% preferably from about 0.1% to about 5% by weight of the total composition.

#### METHOD OF MANUFACTURE

There are many approaches suitable for making the present compositions. If it is desired to form a rinse off treatment, monoalkyl trimethyl ammonium chloride and fatty alcohol are added to hot (70-80°C) water. Then, the mixture is slowly cooled down to room temperature where silicone and other ingredients are added. The compositions may be processed in such a manner that the volatil agent is dispersed in the aqueous phase in particles of from about 1 to about 10 microns.

#### INDUSTRIAL APPLICABILITY

The hair treatment composition of the invention may be formed into a hair treatment preparation, for example a conditioner or conditioning shampoo, a hair rinse or hair treatment spray and the like, according to conventional preparation techniques.

The hair treatment compositions of the present invention are preferably used as a rins on freshly shampoo d hair. The composition is used in an amount of from about 1 g. to about 60 g. preferably from about 2 g. to about 30 g. and is then rinsed from the hair.

The following examples further describe and demonstrate embodiments within the scope of the present invention. The examples are given solely for the purpose of illustration and are not to be construed as limitations of the present invention as many variations thereof are possible without departing from its spirit and scope.

Example	Compound	Yield (%)	mp (°C)
1	1,2,3,4-tetrahydro-6-methyl-5H-benzothiazole	88.2	101-102
2	1,2,3,4-tetrahydro-6-ethyl-5H-benzothiazole	84.2	101-102
3	1,2,3,4-tetrahydro-6-propyl-5H-benzothiazole	82.1	101-102
4	1,2,3,4-tetrahydro-6-isobutyl-5H-benzothiazole	81.5	101-102
5	1,2,3,4-tetrahydro-6-tert-butyl-5H-benzothiazole	80.3	101-102
6	1,2,3,4-tetrahydro-6-cyclohexyl-5H-benzothiazole	79.8	101-102
7	1,2,3,4-tetrahydro-6-(4-methylphenyl)-5H-benzothiazole	78.5	101-102
8	1,2,3,4-tetrahydro-6-(4-ethylphenyl)-5H-benzothiazole	77.2	101-102
9	1,2,3,4-tetrahydro-6-(4-propylphenyl)-5H-benzothiazole	76.1	101-102
10	1,2,3,4-tetrahydro-6-(4-isobutylphenyl)-5H-benzothiazole	75.0	101-102
11	1,2,3,4-tetrahydro-6-(4-tert-butylphenyl)-5H-benzothiazole	74.5	101-102
12	1,2,3,4-tetrahydro-6-(4-cyclohexylphenyl)-5H-benzothiazole	73.8	101-102
13	1,2,3,4-tetrahydro-6-(4-methylphenyl)-5H-benzothiazole	72.9	101-102
14	1,2,3,4-tetrahydro-6-(4-ethylphenyl)-5H-benzothiazole	71.5	101-102
15	1,2,3,4-tetrahydro-6-(4-propylphenyl)-5H-benzothiazole	70.2	101-102
16	1,2,3,4-tetrahydro-6-(4-isobutylphenyl)-5H-benzothiazole	69.1	101-102
17	1,2,3,4-tetrahydro-6-(4-tert-butylphenyl)-5H-benzothiazole	68.0	101-102
18	1,2,3,4-tetrahydro-6-(4-cyclohexylphenyl)-5H-benzothiazole	67.5	101-102
19	1,2,3,4-tetrahydro-6-(4-methylphenyl)-5H-benzothiazole	66.8	101-102
20	1,2,3,4-tetrahydro-6-(4-ethylphenyl)-5H-benzothiazole	65.9	101-102
21	1,2,3,4-tetrahydro-6-(4-propylphenyl)-5H-benzothiazole	64.7	101-102
22	1,2,3,4-tetrahydro-6-(4-isobutylphenyl)-5H-benzothiazole	63.5	101-102
23	1,2,3,4-tetrahydro-6-(4-tert-butylphenyl)-5H-benzothiazole	62.3	101-102
24	1,2,3,4-tetrahydro-6-(4-cyclohexylphenyl)-5H-benzothiazole	61.1	101-102
25	1,2,3,4-tetrahydro-6-(4-methylphenyl)-5H-benzothiazole	60.0	101-102

Prototypes were prepared to compare compositions of the present invention with the prior art technologies via wet detangling and dry combing tests. Unless otherwise indicated, all percentages herein are by weight.

**Prototype formula:**

(wt% finished product)

Raw material	Example1 Comparative	Comparative Example 1	Example 2	
<u>Example 2</u>				
Cetyl Alcohol	2.50	0.50	2.50	
	2.50			
Stearyl Alcohol	4.50	—	4.50	
	4.50			
MBTMAC (80% solid) <sup>1</sup>	2.50	2.50	2.50	—
—				
MLTMAC (35% solid) <sup>2</sup>	—	—	—	
	5.67			
Benzyl alcohol	0.40	0.40	0.40	
	0.40			
Polydimethylsiloxane <sup>3</sup>	4.20	4.20	1.50	—
—				
Cyclomethicone (D5) <sup>4</sup>	—	—	—	
	1.50			
Perfume	0.20	0.20	0.20	
	0.20			
Purified water	85.57	92.07	88.40	
	85.23			
Total	100.00	100.00	100.00	
	100.00			

1) Raw material description/supplier

<sup>1</sup> MBTMAC is Monobehenyl trimethyl ammonium chloride and is commercially available under the trade name of DC-80 from TOHO Chemical.

<sup>2</sup> MLTMAC is Monolauryl trimethyl ammonium chloride and is commercially available under the trade name of LTC-35A from TOHO Chemical.

<sup>3</sup> Polydimethylsiloxane is 15%/85% (wt. basis) mixture of D5 cyclomethicone and dimethicone gum (average molecular weight of about 400,000 to about 600,000).

<sup>4</sup> Cyclomethicone (D5) is Decamethyl cyclopentasiloxane and is commercially available under the trade name of SF1202 from General Electric.

## 2) Prototype making results

Comparative example 1 caused separation, while example 1 became homogenous product. Example 2 and comparative example 2 were progressed to the wet detangling and dry combing test.

## 3) Wet and dry combing test

0.5 cc of product was applied to hair switch (4 g, 20 cm). After rinsing for 30 seconds and squeezing out excess water by finger, the hair switch was combed by plastic comb and combing force was recorded by computer (Wet combing). Then the hair switch was completely dried overnight and was combed again by plastic comb and combing force was recorded by computer (Dry combing). Lower number means better combing performance.

### Wet detangling test

Example 2 showed higher performance in wet detangling than Comparative Example 2.

Prototype	Example 2	Comparative Example 2
Mean	255	338

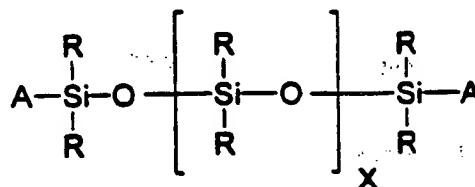
### Dry combing test

Example 2 showed higher performance in dry combing than Comparative Example 2.

Prototype	Example 2	Comparative Example 2
Mean	34	39

**WHAT IS CLAIMED IS:**

1. A hair treatment composition including:
  - (a) from about 0.6% to about 10%, by weight, of a fatty alcohol;
  - (b) from about 0.01% to about 15%, by weight, of a silicone conditioning agent; and
  - (c) from about 0.1% to about 5.0% by weight, of a monoalkyl trimethyl ammonium salt.
2. A hair treatment composition according to claim 1 wherein the silicone conditioning agent is a dispersed, insoluble, non-volatile silicone or mixtures thereof, having a molecular weight of from 200,000 to one million.
3. A hair treatment composition according to claim 1 wherein the monoalkyl trimethyl ammonium salt is a monoalkyl trimethyl ammonium chloride, wherein the alkyl group has at least 14 carbon atoms.
4. A hair treatment composition according to claim 3 wherein the monoalkyl trimethyl ammonium chloride is behenyl trimethyl ammonium chloride.
5. A hair treatment composition according to claim 1 wherein the fatty alcohol has a melting point of higher than 30°C.
6. A hair treatment composition according to claim 5 wherein the fatty alcohol is selected from cetyl alcohol or stearyl alcohol, or mixtures thereof.
7. A hair treatment composition according to claim 1 wherein the silicone conditioning agent has the following formula (I):



(I)

wherein R is alkyl or aryl, X is an integer from about 7 to about 8,000, and A represents any groups which block the end of the silicone chains.

8. A hair treatment composition, according to claim 7, wherein the silicone conditioning agent is a polydimethyl siloxane with a viscosity of from 350 centistokes to about 100,000 centistokes at 25\_C.

## INTERNATIONAL SEARCH REPORT

International application No.  
PCT/US96/13561

## A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) : A61K 7/07

US CL : 424/70.12

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 424/70.12, 70.28, 70.1

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5,334,376 A (ROBBINS ET AL.) 02 August 1994, columns 2-3.	1-8
X	US 4,950,468 A (NAKAMURA ET AL.) 21 August 1990, columns 2-6.	1-8
X	US 4,818,523 A (CLARKE ET AL.) 04 April 1989, columns 4-6.	1-8

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

* Special categories of cited documents:	*T	later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
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Date of the actual completion of the international search

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